HPLC Data Auditing Check Sheet

Method: _____ Laboratory:_____ Rev. 1, 3/04

Hard Copy Data Review	Yes	No	Comments
Proficiency Samples:			
1. Analysis date:			
2. PE successful?			
Calibration:			
1. Standard Information			
-Analysis date:			
-Analyst:			
-Instrument ID:			
-UV Detector			
-Fluorescence Detector			
-Software type:			
-File names:			
2. Quantitation Report and Chromatogram Review			
-Does the lab have adequate hard copy data?			
-Are all standards run the same day/batch? (Check Acquired Times)			
-Is the method update time the same for each file?			
-Is the chromatogram info the same as the quant. reports (i.e. same file names, acquisition times, method update times, print time)?			

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Method: _____ Laboratory:_____ Rev. 1, 3/04

-Is the chromatogram printed using a scale that is visible?			
-Do the standards have the proper sensitivity?			
-Do the standard peaks have acceptable separation?			
-No significant contamination?			
-Are the peaks properly ID'd?			
-Do the peak responses on the quant. reports match those of the calibration summary report (hand calculate a few-especially manual integrations)?			
-Do the calibration levels support the laboratory's reporting levels (check cal. level vs. final report of sample vs. MDLs)?			
3. Calibration Method Information			
-Quantitation method file name:			
-Calibration type (i.e. linear, RF, etc.):			
-Same for all compounds?			
-Was the calibration criteria met for each compound (i.e. RSDs)?			
-"force thru the origin"?			
-Were data points eliminated from the calibration?			
-If yes, why?			
-Was this done appropriately?			
Attach photo copy documentation of any areas of concern			

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Sample Information:			
-Sample date/time (from COC):			
-Were the samples properly preserved?			
- Does the final report have the AZ License noted?			
Sample Preparation Procedures:			
-Extraction method:			
-Extraction date/time:			
-Did the sample meet the extraction hold time?			
-Is the extraction documentation correct and complete?			
- Did the extraction need clean up (EPA 3630)?			
-Was the extraction acceptable (refer to check sheets or hand notes)?			
Attach photo copy documentation of any areas of concern			
Sample Analysis:			
-Sample ID:			
-Analysis date/time:			
-Was the sample hold time met?			
-Was the proper QC run with the sample batch?			
-Was the QC at the proper concentrations?			
-Was the appropriate QC (including tune if MS) criteria met?			

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- What are the flow rates?			
-Do all low level QC checks have adequate sensitivity?			
-Does the hard copy data correspond to the sequence report?			
-Are there any major breaks in the acquisition times?			
-Do all the samples/QC in the batch have the same method update time?			
-Do all chromatograms have corresponding information to the respective Quant Report (i.e. same file names, acquisition times, method update times, same RTs, print time)?			
-Are the response factors of the samples the same as from the calibration (calculate a few)?			
-Are the chromatograms printed using a scale that is visible?			
-Do all samples/QC in the batch have adequate peak separation?			
-No significant contamination or matrix interference?			
-Are the peaks properly ID'd?			
-Are all the peaks integrations appropriate and consistent?			
-Do the analytical results on the Quant Report match those on the final report?			
Attach photo copy documentation of any areas of concern			

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Method: _____ Laboratory:_____ Rev. 1, 3/04

Laboratory Review	Yes	No	Comments
-Was the analyst(s) available for interviewing?			
-Did the analyst(s) provide adequate response to the concerns found from the hard copy data review?			
-Was the analyst(s) following proper procedure? -If no, see notes or check sheets. -If no, is SOP correct? -If no, is the QAP correct?			
-Did the lab have the proper equipment and instrumentation?			
-Did the lab have the proper reagents?			
-Did the lab have adequate documentation such as run logs, maintenance logs, temperature logs and standard logs?			
- Are the eluent bottles labeled?			
Electronic Data Review:	Yes	No	Comments
1. Mint Miner Review (If Applicable) -Are any problems identified?			
In-Lab Review:			
2. High and low standard			
-Does the low standard have acceptable sensitivity			
-Do all the compound peaks have adequate separation?			

HPLC Data Auditing Check Sheet Method: _____ Laboratory:_____ Rev. 1, 3/04			
-Do all the compound peaks have appropriate and consistent integration?			
3. Initial CCV			
-Do all the peaks have adequate sensitivity?			
-Do all the peaks have adequate separation?			
-Do all the peaks have appropriate and consistent integration?			
-Can the laboratory reprint a Quant Report and chromatogram that matches the hard copy?			
-If yes, Attach.			
-If no, why?			
4. Other electronic data concerns (Identified in the hard copy review):			
Attach photo copy documentation of any areas of concern			
Training: -If significant problems are noted above, do the analyst's training files show that they were properly trained?			

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
531.1	9.3.1 & 9.3.2	ICAL	3 pts.	<20% RSD		

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
Rev. 3.0 Carbamates (Fluorescence)	9.3.3	DAILY	beginning & end of run, two different concentrations	±20%		
	10.6.1, 10.3.2 & Table 2	LFB (LCS)	one per set or 20 samples	Table 2, R±30%		
	10.7.1	MS	5 %	same as LFB		
	11.2.3	Mobile Phase	Methanol/water (400 ul sample injection)			
547 Glyphosate July 1990 (Fluorescence)	9.2 & 9.3	ICAL	3 pts	<10% RSD		
	9.4	DAILY	beg & end, different conc.	±20%		
	10.5 & 10.3.2	LFB	one per set or every 24 hr.	Table 2 R±30%		
	10.6.1 & 10.6.2	MS	10% or one per set	Table 2 R ±30%		
	7.1.1 & Table 1 (sec. 10.4 - can modify conditions)	Mobile Phase	0.005 M KH2PO4 in 960 ml H2O/ 40 ml MeOH, adjust to pH 1.9. Calcium hydrochlorite & OPA for post column Derivatization made daily, 200ul sample injection			
549 Diquat & Paraquat Rev 1.0 August 1992 (UV)	9.3	ICAL	3 pts Diquat @ 308nm Paraquat @ 257nm	prepare curve		
	9.4	DAILY	beg & end, different conc.	±20%		
	10.5 & 10.3.2	LFB	one per set/24hr	Table 2 R±30%		
	10.6	MS	10%	same as LFB		

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
	7.16	Mobile Phase	3 g 1-hexanesulfonic acid, sodium salt, 13.5 ml ortho-phosphoric acid, 10.3 ml diethylamine in 1 L water			
549.1 Diquat & Paraquat Rev. 1.0 August 1992 (UV)	10.3	ICAL	3 pts Diquat @ 308nm Paraquat @ 257nm	prepare curve		
	10.4	DAILY	beg. & end. different conc.	±20%		
	9.5	LFB	1 per set/ 24 hr	Table 2 R±30%		
	9.6	MS	10% or one per set	same as LFB		
	7.16	Mobile Phase	3 g 1-hexanesulfonic acid, sodium salt, 13.5 ml orthophosphoric acid, 10.3 ml diethylamine in 1 L water			
550 &550.1 PAH (method sections are the same) July 1990	9.2	ICAL	3 pts			
	9.4	DAILY	beg. & end different conc.			
	10.5 &10.3.2	LFB	one per set/24hr			
	10.6	MS	10% one per set			
	Table 1	Mobile Phase	Acetonitrile and water			
553 Benzidines & Nitropesticides LC/MS Rev 1.1 August 1992	7.12 & 10.2.9	ICAL	6 pts	<20%		
	Tune: 10.3.1 Cal: 10.3.2, 10.3.4 & 10.3.5	DAILY	Tune:use DFTPPO every 8 hours Cal:mid level every 8 hrs.	Tune: Table 1 Cal: ±20% area of Ical Std. & ±20% of true value		
	9.5 & 9.3.3	LFB	one per sample set	70-130%		

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
	9.6, 9.1 & 9.3.3	MS	regularly	70-130%		
	7.13	Mobile Phase	75/25 water/ACN with Ammonium Acetate @ 0.01 M			
	7.1, 9.3.3	Surrogate	70-130%			
554 Carbonyl Rev 1.0 August 1992	10.2	ICAL	5 pts. External only, derivatize & extract the standards	prepare curve		
	10.2.2.2	DAILY	each day	±10 %		
	9.4	LFB	one per 20 sample or per 24hr	lab sets		
	9.5, 9.4	MS	10% or per sample set	same as LFB limits established		
	10.1	Mobile Phase	MeOH/water			
		section 10.1 “Establish the HPLC operating parameters to completely separate peaks”				
555 Chlorinated Acids Rev 1.0 August 1992 UV detector	10.1 & 10.2	ICAL	External cal only. Minimum 3 standards	20% RSD or curve		
	10.2.3	DAILY	each analysis day. Recommend end of day	±25%		
	9.5.1, 9.5.2 & 9.3.2, Table 2	LFB	one per 20 samples or every 24 hr, whichever is greater	R ±30%		

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
	9.6.1, 9.6.2	MS	10%	if no contamination, same as LFB. If cont. Use formula in section 9.6.2		
	6.4.1, 9.4	Mobile Phase	0.025 M H_3PO_4 & Acetonitrile, gradient, but analyst permitted to change columns, conditions and detectors			
		Confirmation column required (sec.6.4). No unresolved peaks in the same mixture (sec. 10.1) must separate all analytes between primary & confirmation columns.				
610 PAH UV and/or Fluorescence detector Note:GC can also be done for this method	7.2 external 7.3 internal	ICAL	3 points	RF<10% RSD		
	7.4	DAILY	each working day	±15%		
	8.4	LFB	when MS/MSD fails	Table 3		
	8.3	MS/MSD	10% of samples	Table 3, column P		
	12.2 &Table 1	Mobile Phase	water and acetonitrile - gradient to 100% ACN			
8310 PAH	8000B, section 7.4 & 7.5	ICAL	5 points for linear 6 pts for quadaratic 7 for third order (polynomial)	<20%RSD to use average RF Cannot force 2nd or third order through zero		
	8000B, section 7.7 for average, 7.7.1for linear, 7.7.2 for non-linear	DAILY	beginning & end (8.2.2) of each twelve hour shift. And every ten samples recommended (7.7.6)	±15% response, concentration or drift		

Method/Analyte	Method Reference	QC	Frequency	Limits	Lab SOP	COMMENTS
	8000B, section 8.5	LFB	one per batch up to 20 samples extracted together	in-house. Should be ~70-130%		
	8000B, section 8.5	MS/MSD	same as above	same		
	8000B, section 8.6	Surrogate	each sample	in-house (8.7)		
	8310, section 7.2	Mobile Phase	water/Acetonitrile			
8330 Explosives	8000B, section 7.4 & 7.5	ICAL	5 points for linear 6 pts for quadaratic 7 for third order (polynomial)	<20%RSD to use average RF Cannot force 2nd or third order through zero		
	8330, section 7.3.3	DAILY	beginning & end of each group of 10 samples and midway through sequence	±15% response, concentration or drift		
	8000B, section 8.5	LFB	one per batch up to 20 samples extracted together	in-house. Should be ~70-130%		
	8000B, section 8.5	MS/MSD	same as above	same		
	8000B, section 8.6	Surrogate	each sample	in-house (8.7)		
	8330, section 7.2	Mobile Phase	50/50 methanol/water (recommended)			